Figure 2I

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Supporting Information for Szyperski *et al.* (2002) *Proc. Natl. Acad. Sci. USA* **99** (12), 8009–8014. (10.1073/pnas.122224599).

Supporting Figure 10

Fig. 10. Experimental scheme for the 3D HCCH-TOCSY experiment. Rectangular 90° and 180° pulses are indicated by thin and thick vertical bars, respectively, and phases are indicated above the pulses. Where no rf phase is marked, the pulse is applied along *x*. The scaling factor k for ^{1}H chemical shift evolution during t_1 is set to 1.0. The high power 90° pulse lengths were: 5.8 ms for ^{1}H and 15.4 ms for ^{13}C, and 38 ms for ^{15}N. ^{13}C decoupling during t_1(^{1}H) is achieved using a (90_x 180_y 90_x) composite pulse. The lengths of the ^{1}H spin-lock purge pulses are: first SL_x, 5.7 ms; second SL_x, 0.9 ms; SL_y, 4.3 ms. SEDUCE is used for decoupling of ^{13}CO during t_1 and t_2 (rf field strength = 1 kHz), and GARP is employed for decoupling of ^{13}C during acquisition (rf = 2.5 kHz). The ^{1}H rf carrier is placed at the position of the solvent line at 0 ppm before the start of the first semiconstant-time ^{1}H evolution period, and then switched to the water line at 4.78 ppm after the second 90° ^{1}H pulse. The $^{13}C^a$ and ^{15}N rf carriers are set to 38 and 120.9 ppm, respectively. The length of ^{13}C spin-lock purge pulses denoted SL_x are of 2 ms duration. ^{13}C isotropic mixing is accomplished using the DIPSI-2 scheme (rf = 8.5 kHz). The duration and strengths of the pulsed z-field gradients (PFGs) are: G1 (100 ms, 16 G/cm); G2 (2 ms, 15 G/cm); G3 (300 ms, 8 G/cm); G4 (500 ms, 30 G/cm); G5 (100 ms, 16 G/cm). All PFG pulses are of rectangular shape. A recovery delay of at least 100 ms duration is inserted between a PFG pulse and an rf pulse. The delays are: t_1 = 850 ms, t_2 = 3.2 ms. ^{1}H- frequency labeling in t_1 is achieved in a semiconstant-time fashion with t_1^a (0) = 1.7 ms, t_1^b (0) = 1 ms, t_1^c (0) = 1.701 ms, Dt_1^a = 33.3 ms, Dt_1^b = 19.3 ms, and Dt_1^c = -14 ms. ^{13}C- frequency labeling in t_2 is achieved in a semiconstant-time fashion with t_2^a (0) = 1120 ms, t_2^b (0) = 62.5 ms, t_2^c (0) = 995 ms, Dt_2^a = 160 ms, Dt_2^b = 125 ms, and Dt_2^c = -35 ms. These delays ensure that a 90° first-order phase correction is obtained along w_2(^{13}C). The fractional increases of the semiconstant-time period in t_1 equals to l = 1 + Dt_2^c / Dt_2^a = 0.58, and in t_2 equals to l = 1 + Dt_2^c / Dt_2^a = 0.78. Phase cycling: $f_1 = x$; $f_2 = x, -x$; $f_3 = x$; $f_4 = 2(x), 2(-x)$; f_5(receiver) = $x, -x$. Quadrature detection in t_1(^{13}C/^{1}H) and t_2(^{13}C) is accomplished by altering the phases f_2 and f_3, respectively, according to States-TPPI. For acquisition of central peaks derived from ^{13}C steady state magnetization, a second data set with $f_1 = -x$ is collected. The sum and the difference of the two resulting data sets generate subspectra II and I, respectively, containing the central peaks and peak pairs.

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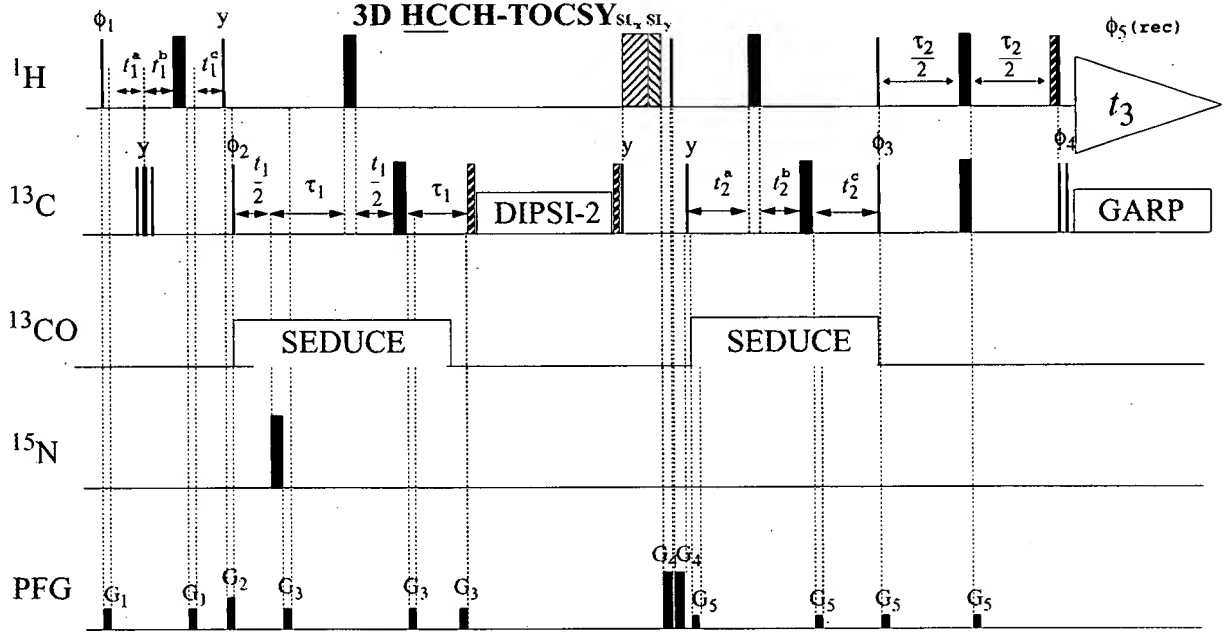
3D HCCH-TOCSY
ϕ_1
y
t_1^a t_1^b t_1^c
ϕ_2
$\frac{t_1}{2}$
τ_1
DIPSI-2
$\frac{\tau_2}{2}$
ϕ_3
t_2^a t_2^b t_2^c
ϕ_4
ϕ_5 (rec)
t_3
GARP
SEDUCE
SEDUCE
^{1}H
^{13}C
^{13}CO
^{15}N
PFG
G_1 G_1 G_2 G_3 G_3 G_3 G_4 G_4 G_5 G_5 G_5 G_5

Figure 10